



Office de la propriété  
intellectuelle  
du Canada

Un organisme  
d'Industrie Canada

Canadian  
Intellectual Property  
Office

An Agency of  
Industry Canada

PCT/CA 2004/001888

01 DECEMBER 2004 01.12.04

*Bureau canadien  
des brevets  
Certification*

La présente atteste que les documents  
ci-joints, dont la liste figure ci-dessous,  
sont des copies authentiques des docu-  
ments déposés au Bureau des brevets.

*Canadian Patent  
Office  
Certification*

This is to certify that the documents  
attached hereto and identified below are  
true copies of the documents on file in  
the Patent Office.

REC'D 28 DEC 2004

WIPO

PCT

Specification and Drawings, as originally filed, with Application for Patent Serial No:  
2,447,098, on October 28, 2003, by **CENTRE DE RECHERCHE INDUSTRIELLE DU  
QUÉBEC**, assignee of Mokhtar Beanoudia, Luc Laperrière, Pierre Bédard, Claude  
Lejeune, Céline Leduc and Claude Daneault, for "Method for Estimating an Optimal  
Dosage of Bleaching Agent to be Mixed with Wood Chips".

**PRIORITY  
DOCUMENT**  
SUBMITTED OR TRANSMITTED IN  
COMPLIANCE WITH RULE 17.1(a) OR (b)

Agent certificateur/Certifying Officer

December 1, 2004

Date

Canada

(CIPD 68)  
31-03-04

OPIC



CIPO

**BEST AVAILABLE COPY**

**Abstract of the disclosure**

A method for estimating an optimal dosage of bleaching agent to be mixed with wood chips to be fed to a process for producing pulp characterized by a required brightness value. According to a set of wood chip properties characterizing the wood chips as estimated by a measurement system, corresponding wood chip properties data are fed at the inputs of a neural network 12, as well as an initial dosage value of the bleaching agent. The neural network generates a predicted brightness value for pulp to produce from the inspected wood chips. The brightness predicted value is compared with the required brightness value to generate error data, which is used to optimize the bleaching agent dosage value by minimizing the error data. Prediction, comparison and optimization steps are repeated with the optimized bleaching agent dosage value until the brightness predicted value substantially reaches the required brightness value, to estimate the optimal bleaching agent dosage.

## **METHOD FOR ESTIMATING AN OPTIMAL DOSAGE OF BLEACHING AGENT TO BE MIXED WITH WOOD CHIPS**

### **Field of the invention**

5       The present invention relates to the field of pulp and paper process automation, and more particularly to methods for estimating optimal dosage of bleaching agent to be mixed with wood chips to be fed to a process for producing pulp characterized by a required brightness value.

### **Background of invention**

10       Thermomechanical pulp properties and quality are influenced by two types of variables: feed material (chips) and process (refiner). Over the years, many researchers have underscored the impact of the stability of the refiner operation for the production of constant pulp quality, as mentioned by Strand, B. C. in "The Effect of Refiner Variation on Pulp Quality",  
15   International Mechanical Pulping Conference, Proceedings, 125-130 (1995). However, variations of the process itself are mainly related to variations in the raw material feeding the system as, mentioned by Wood, J. A. in "Chip Quality Effects in Mechanical Pulping – a Selected Review", 1996 TAPPI  
20   Pulping Conference, Proceedings, 491-497 (1996). In particular, pulp brightness is considered as an important quality requirement, as discussed by Dence, C. W. et al. in "Pulp Bleaching – Principles and Practice", TAPPI Press, 457-490 (1996).

25       An object of the method according to the invention is to model the relationship between the quality of the chips feeding the process with an important pulp and paper resulting property, namely pulp brightness. In particular, the model is used to evaluate the minimum charge of peroxide required to reach certain level of pulp brightness according to possible chips properties fluctuations, in order to minimize the cost and environmental impact of the bleaching operation.

**Summary of invention**

According to the above mentioned object, there is provided a method for estimating an optimal dosage of bleaching agent to be mixed with wood chips to be fed to a process for producing pulp characterized by a required brightness value. The method comprises the steps of: i) estimating a set of wood chip properties characterizing said wood chips to generate corresponding wood chip properties data, said set including reflectance-related properties; ii) providing an initial dosage value of said bleaching agent; iii) feeding said wood chip properties and said bleaching agent dosage value at corresponding inputs of a neural network previously trained according to experimentally obtained data on said wood chip properties and on dosage of said bleaching agent, to generate predicted brightness value for pulp to produce from said wood chips; iv) comparing said brightness predicted value with said required brightness value to generate error data; v) optimizing said bleaching agent dosage value to minimize said error data; and vi) repeating said steps iii) to v) with said optimized bleaching agent dosage value until said brightness predicted value substantially reaches said required brightness value, to estimate said optimal bleaching agent dosage.

**Brief description of the drawings**

The method according to the present invention will be described in detail with reference to the accompanying drawings in which:

Fig. 1 is a graph showing relative importance index of independent variables according to PLS analysis;

Fig. 2 is a graph showing coefficient of correlation for dependent variables by PLS analysis;

Fig. 3 is a graph representing observed and predicted values for ISO brightness; and

Fig. 4 is a block diagram of a neural network-based model that can be used to carry out the method according to the invention.

### Detailed description of the preferred embodiment

In order to define the parameters used for the model, two sets of experiments corresponding to two different blocks were performed.

In the first block, a potential mix of four species, black spruce, balsam fir, jack pine and white birch, was studied. The last two species were chosen because they represent a potential source of new resources. The trees have been selected, cut, barked and chipped in order to obtain standard chips with known and controlled age. In fall, outdoor stacks of each species of chips were prepared. During the following 12 months, six samples were selected in order to conduct the experimental plan for chips aging as described in table 1.

Test number	Spruce%	Balsam fir%	Jack pine%	Birch%
1	0	0,2	0,4	0,4
2	1	0	0	0
3	0	1	0	0
4	0,6	0	0	0,4
5	0	0,6	0,4	0
6	0,6	0	0,4	0
7	0	0,6	0	0,4
8	0,2	0	0,4	0,4
Following are repetitions for experimental error determination				
9	1	0	0	0
10	0	1	0	0
Following are additional tests				
11	0	0	1	0
12	0	0	0	1

Table 1

In each sample, the experiments for 100% black spruce and 100% balsam fir were repeated twice in order to evaluate the experimental error (14 runs in each sample). The six samples allow to evaluate the evolution of the quality, i.e. degradation, of the chips in time. This degradation is highly dependent on storage temperature. The first four samples were evaluated at an interval of three weeks. After that, there has been a longer waiting time. It was noticed that the winter degradation of each stack was extremely slow.

The second block of experiments was used to investigate the effects of other important variables regarding pulp quality. This second block of experiments has been conducted with four variables: species (black spruce, balsam fir), density (high, low), initial dryness of the chips (fresh, dry), and thickness of the chips (0-4 mm, 4-8 mm). Table 2 describes the experiments for chips aging that were conducted in this second block.

<b>INITIAL CHIPS</b>		
	<b>Large chips (4-8 mm)</b>	<b>Small chips 0-4 mm</b>
Spruce at low density	Test no. 1	Test no. 2
Balsam fir at low density	Test no. 3	Test no.4
Spruce at high density	Tests no.5 and 6	Test no.7
Balsam fir at high density	Test no. 8	Tests no. 9 and 10
<b>Aged chips (dryness of 75%)</b>		
	<b>Large chips (4-8 mm)</b>	<b>Small chips 0-4 mm</b>
Spruce at low density	Test no. 11	Tests no.12 and 13
Balsam fir at low density	Tests no.14 and 15	Test no. 16
Spruce at high density	Test no. 17	Test no. 18
Balsam fir at high density	Test no. 19	Test no. 20

**Table 2**

- 5 Refining was conducted on a pilot unit Metso CD-300. Each sample was washed and refined in two stages. The first one was conducted at a temperature of 128°C and the second one at atmospheric pressure. For each experiment, pulps with a freeness ranging from 200 to 150 mL were selected for further peroxide bleaching, which fundamental principles are briefly
- 10 described next.



It is generally accepted that the active mechanism in chromophore elimination with hydrogen peroxide as bleaching agent involves the perhydroxyl ion  $\text{OOH}^-$ . As taught by Sundholm, J. in "Papermaking Science and Technology – Mechanical Pulping", Finnish Pulp and Paper Research Institute, 313-345 (1999), hydrogen peroxide bleaching is therefore performed in alkaline systems to produce the active ion according to the following equation:



The formation of the perhydroxyl anion can be enhanced by increasing the pH or by increasing the temperature. Hydrogen peroxide readily decomposes under bleaching conditions according to the following equation:



Sodium silicate and magnesium silicate are normally added to the bleach liquor to stabilize peroxide. Transition metals ions like iron, manganese and copper catalyze peroxide decomposition. In order to prevent that, before bleaching with peroxide, the pulp was pretreated with 0,2% of DTPA. The pretreatment of the pulp was done at 60°C, 15 minutes and 3 % of consistency.

Different concentrations of hydrogen peroxide varying from 1 to 5% (O.D. basis) were tested for bleaching the different pulp. Table 3 describes the experimental conditions used for the peroxide bleaching of the pretreated pulps.



Parameters	P1	P2	P3	P5
Temperature, °C	70	70	70	70
Retention time, min	180	180	180	180
Consistency, %	12	12	12	12
Sodium silicate, %	3,00	3,00	3,00	3,00
Magnesium sulfate, %	0,05	0,05	0,05	0,05
Total Alkali Ratio	2,00	1,20	0,90	0,80
Sodium hydroxyde, %	1,66	2,06	2,36	3,66
Hydrogen peroxide, %	1,00	2,00	3,00	5,00

**Table 3**

where:

$$5 \quad \text{Total Alkali Ratio} = \frac{\% \text{ Hydrogen Peroxide}}{\% \text{ OH- given by sodium silicate and sodium hydroxide}} \quad (3)$$

Bleaching was conducted at 70°C, 180 minutes and 12 % of consistency. The bleaching liquor was composed of 3,00% of sodium silicate, 0,05% of magnesium sulfate, hydrogen peroxide and sodium hydroxide. After the  
 10 bleaching step, the pulp was diluted at 1% of consistency and neutralized with sodium metabisulfite at pH 5,5. A volume of the bleaching liquor was kept to measure the residual peroxide by an iodometric dosage. Optical properties such as ISO brightness and color coordinates ( $L^*$ ,  $a^*$ ,  $b^*$ ) have been measured according to Paptac standard.

15 Chips of the eighty four (84) runs in block 1 and twenty (20) runs in block 2 were systematically analyzed using a wood chip optical inspection apparatus known as CMS-100 chip management system commercially

available from the present assignee, Centre de Recherche Industrielle du Québec (Ste-foy, Canada), for measuring a number of optical properties as well as moisture content. Such wood chip inspection apparatus is described in U.S. Patent no. 6,175,092 B1 issued on January 16, 2001 to the present assignee. Such multi-sensor system includes main and optional auxiliary sensors able to characterize wood chips online. The main sensors include artificial vision sensor (an RGB color camera) and near infrared sensor to measure chip brightness and moisture content. Auxiliary sensors such as a distance sensor and an air conditions sensor to measure air temperature and relative humidity may be advantageously used. They provide information that extends measurements of the main sensor to stabilize the system (for example, variations of the camera measuring distance will influence the chip brightness measurement). The system will work on frozen and non-frozen wood chips, and it used for predicting bleach charges or dosage based on chip quality for use as a bleach control method or system. The correlation between some chip properties and its possible application in bleach control is discussed by Ding, F. et al. in "Economizing the Bleaching Agent Consumption by Controlling Wood Chip Brightness", *Control System 2002, Proceedings*, June 3-5, Stockholm, Sweden, 205-209 (2002). The most relevant wood chips properties measurements for the purpose of the present invention are described next.

A first measurement relates to chip luminance, wherein the brightness of black is defined as zero and the brightness of white as 150. The RGB colour camera of the system is calibrated by a color checker made of black and white paperboard. The wood chip color is between white and black, so its brightness is between 0 and 150. A second measurement relates to chip average moisture content. The system includes a near infrared sensor that is used to measure surface moisture content of wood chips, without any non-contact therewith. A phenomenological model may be used to calculate the average moisture content from surface moisture content, as described by

Ding, F. et al. in "Economizing the Bleaching Agent Consumption by Controlling Wood Chip Brightness", *Control System 2002, Proceedings*, June 3-5, Stockholm, Sweden, 205-209 (2002). Other measurements may be obtained from various further sensors, generating a large amount of data categorized in many different variables. According to the present preferred embodiment, a number of four (4) other measurements are considered, namely the image "H", "S" and "L" parameters, as well as a chip average size estimation, which may be obtained using a surface-based, chip size classifier such as disclosed by Ding, F. et al. in "Wood Chip Physical Quality Definition and Measurement", *IMPC Proceedings*, June 2-5, Québec, Canada, 367-373 (2003).

The database resulting from the various experiments gives rise to three (3) types of variables: chip properties coming from the measuring system, operational parameters of the TMP and bleach processes, and pulp quality characteristics. Overall, the database used contained a large number (n=178) of variables distributed over a corresponding number of columns. Because all (104) runs for both blocks produced pulps which were bleached at four (4) different peroxide charges, the database also contains four times (416) runs distributed over a corresponding number of lines. In order to capture possible system measurements errors, the database contained many repeated measurements for the same chips, leading a final database containing a still greater number (506) of data lines. In the following sections, the techniques that are preferably used to screen the columns of data to a reasonable amount of most relevant variables and to use the lines for neural network training will be explained. Both techniques are done with the objective of obtaining a good enough pulp brightness model that could be used in a brightness control strategy.

The data screening to perform the selection of the independent variables which have an effect on the dependent variables that have been measured is preferably done using known PLS (Projection on a Latent

Structure) modeling. Fig. 1 presents the independent variables that have been chosen according their relative importance index. As expected, the parameters which have most impact and are correlated to dependent variables are the concentration of sodium hydroxide (NaOH) and the concentration of hydrogen peroxide (PEROA). After that, the variables Co\_moy (chip size), MDH (average of H), MMLC (average of luminance), MDS (average of S), MDL (average of L) and MSURFM (average of the surface moisture) also contribute to a lesser extent to the bleached pulp properties response.

The correlation coefficients for each dependent variable are presented in Fig. 2. The value R2 shows the correlation for the dependent variables. It is an indication of how well the model can fit the experimental data. The value Q2 shows the correlation of the interpolated responses, i.e. predictions not part of the experimental data. The graph shows that the model is adequate to predict ISO brightness [ISOB] (coefficient of correlation of 0,88), color coordinates L\* [LB] (coefficient of correlation of 0,92) and a\* [AB] (coefficient of correlation of 0,90), and residual peroxide [PEROR] (coefficient of correlation of 0,83). As for the color coordinate b\* [BB], the coefficient of correlation is only 0,60. We also note that chemical properties such as MEXT. (extractives) and AGR (fatty and resinic acids) are difficult to correlate (coefficients of correlation of 0,33 and 0,26 are respectively obtained).

Fig. 3 presents observed and predicted values for the ISO brightness. These results show that the model is able to predict adequate values for this optical property. Brightness ranging from 43.79% to 80.2% were measured on the bleached pulps.

A neural network-based model that can be used to carry out the method according to the invention will now be described in reference to Fig. 4. The model generated designated at 10 includes a neural network 12 that was previously trained according go to the experimentally obtained data on

wood chip properties and on dosage of said bleaching agent as described above, i.e. over the nine (9) remaining database columns consisting of eight (8) inputs identified by PLS method as shown in Fig. 2, and one output, namely pulp brightness as shown in Fig. 3. Such known neural network and associated training approach are discussed by Laperrière L. et al. in "Modeling and simulation of pulp and paper quality. After a few unsuccessful training trials, it was noticed that the input NaOH is always a ratio of the input H<sub>2</sub>O<sub>2</sub>, so it was eliminated from the training set. Out of the available (506) training lines, a selected number (96) were removed (about 20%) and injected back to the trained network for validation. Different sets of the removed 20% were tested and gave similar results. The final configuration was a 7-5-1 neural network (7 inputs, 5 hidden neurons and 1 output) as designated at 12 in Fig. 4. Training was stopped after an average absolute mean error of 5% was reached between the neural network prediction and the training output brightness value for each of the 506 lines. The value of 5% was chosen by taking two factors into consideration: 1) reliability of the output measurements: the experimental error related to the brightness value is about 3%, i.e.  $\pm 0.5$  brightness points in the experimental span of 43.79 to 80.2 measured brightness; and 2) reliability of the input measurements: calibration errors may encourage an increase of the training error. The training results of the final network, in terms of the connection weights between each of its constituting neurons, were imported into the neural network 12 of model 10, in the form of a computer program that can be implemented in a microcomputer by any person skilled in the art using well known programming tools. Such program is able to simulate brightness prediction based on the seven (7) chosen inputs, namely reflectance-related properties of wood chips that are Luminance, M, H, S, L and chip size from measurement system 14, and bleaching agent dosage (peroxide charge) value used by the bleaching unit as represented at 16. Optionally, unmodeled disturbances may also be applied to the neural network at input 17.



In operation, according to the set of wood chip properties characterizing the wood chips as estimated by the measurement system 14, corresponding wood chip properties data are fed at respective inputs 18 of neural network 12, as well as an initial dosage value of the bleaching agent (peroxide) at further input 20. In turn, the neural network 12 generates at output 22 thereof, a predicted brightness value for pulp to produce from the inspected wood chips. Then, the brightness predicted value is compared with the required brightness value to generate error data, as indicated at node 24. In turn, the error data is used by an optimization module 26, which optimizes the bleaching agent dosage value to minimize the error data. Finally, the above prediction, comparison and optimization steps are repeated with the optimized bleaching agent dosage value until the brightness predicted value substantially reaches the required brightness value, to estimate the optimal bleaching agent dosage. In other words, the peroxide charge is tuned to minimize the error, while maintaining constant chip properties, and an optimization loops is performed in model 10 for several iterations before it reaches the peroxide charge that meets the required brightness value or setpoint according to the neural network model prediction. When this optimal value has been found, it can be sent back to the actual process for corrective action on a control valve provided on bleaching unit 26. Such control strategy assumes that the time taken for the optimization to take place is less than the frequency at which brightness setpoints will be modified, which is a reasonable assumption.

Because the brightness prediction is based upon variables that are algorithmic transformations of camera signals, a first simulation was designed in which the neural network model was used in conjunction with an optimizer that would find the best combination of measurement system input variables that would give the best achievable brightness. Simulation results are shown in table 4.

Variable	Min. experimental value	Max. experimental value
Luminance	13.23	57.93
Moisture	19.54	70.34
Size	16.49	21.39
H	19.29	224.44
S	82.08	192.89
L	9.17	66.85
H2O2	0.00	5.00

Table 4

There are two main observations from this result. First, for optimal  
5 brightness all independent variables are either at the minimum or maximum  
values or their respective span. This means that the hyper surface for which  
a minimum was found slants towards an intersection of the constraint hyper  
planes corresponding to the maximum or minimum values of each  
independent variable. This also means there is a well defined combination  
10 for maximum brightness (the optimal combination was consistently  
reproduced for many different simulation trials). Second, we see that five  
(5) of the six (6) system measurements give the best pulp brightness when  
they are at their higher values, except for the "H" parameter (lowest value).

Turning back to Fig.1, the sensitivity of output properties with respect  
15 to some of the chosen inputs is shown. Because this result was obtained  
from a PLS model and the use a neural network model is contemplated,  
another set of simulations was run to verify if the sensitivity of the sole pulp  
brightness to each independent variable would be similar. Table 5 shows a  
series of tests where each variable was given sinusoidal swings of its value  
20 over its total experimental span as shown in table 4, while maintaining other  
variables at their central values, to show brightness sensitivity to the  
independent variables.



Variable	Min. brightness	Max. brightness	% change in brightness
Luminance	63.55	68.02	12.3
Moisture	64.52	67.01	6.8
Size	64.60	67.48	7.9
H	63.81	67.71	10.7
S	63.99	67.69	10.1
L	65.18	66.52	3.6
H2O2	49.57	70.83	58.4

**Table 5**

- 5 It turns out that peroxide has a predominant effect. In fact, the peroxide charge fixes the brightness level and changes in the chip properties simply add small variations around the level attained. Every system measurement variable, when bumped independently within its full span, contributes to small percentage of change around the brightness level dictated by the peroxide charge.
- 10

In order to illustrate brightness control feature, a first set of simulation results is shown in table 6, representing the effect of chip quality on peroxide charges to achieve different brightness setpoints.

<b>Brightness setpoint (%)</b>	<b>Peroxyde charge (%) average quality chips</b>	<b>Peroxyde charge (%) best theoretical chips</b>	<b>Peroxyde charge (%) best experimental chips</b>
55	0.77	0.0	0.15
60	1.41	0.0	0.76
65	2.22	0.35	1.48
70	4.12	1.21	2.92
71	4.99	1.48	3.54
75	Unachievable (max 71%)	4.96	Unachievable (max 72%)

**Table 6**

5 All measurement system parameters (chip properties) were maintained at their average value and brightness setpoint was bumped from 55 to 75 by increments of (five) 5 points. For these "average" chips, one can see that a 38% increase in peroxide (from 2.22 to 4.12%) is required to increase the brightness level from 65 to 70 points (13.7%), and that a further 17.6% increase (from 4.12 to 5%) is required to gain only 1 brightness point (from 70 to the maximum achievable 71) Doing the same thing with the theoretical best possible chips as per table 4, one can note that no peroxide is required until a brightness setpoint close to 65 is desired. Also, a 71 brightness is achievable with only 1.48% peroxide. Finally, further gains in brightness points from 71 to 75 are only obtained at a high peroxide cost (from 1.48 to 4.96%). Because one cannot assume that chips with such properties actually exist, the chip properties contained in the above mentioned database were also used, which returned the best brightness value

at 72.46. In this case, the better chip properties still reduce peroxide consumption for the same brightness level, but to a lesser extent.

When using the method according to the invention, the same brightness setpoint can be achieved at lower bleaching agent charges when the chip quality increases. The method may be useful to assist chip management in the mill, or in the context of internal model control (IMC) or model predictive control (MPC) strategies. It is to be understood that dosage of other bleaching agents such as hydrosulfites may also be performed with the method of the invention.

We claim :

1. A method for estimating an optimal dosage of bleaching agent to be mixed with wood chips to be fed to a process for producing pulp  
5 characterized by a required brightness value, said method comprising the steps of:
  - i) estimating a set of wood chip properties characterizing said wood chips to generate corresponding wood chip properties data, said set including reflectance-related properties;
  - 10 ii) providing an initial dosage value of said bleaching agent;
  - iii) feeding said wood chip properties and said bleaching agent dosage value at corresponding inputs of a neural network previously trained according to experimentally obtained data on said wood chip properties and on dosage of said bleaching agent, to generate predicted brightness value for  
15 pulp to produce from said wood chips;
  - iv) comparing said brightness predicted value with said required brightness value to generate error data;
  - v) optimizing said bleaching agent dosage value to minimize said error data; and
  - 20 vi) repeating said steps iii) to v) with said optimized bleaching agent dosage value until said brightness predicted value substantially reaches said required brightness value, to estimate said optimal bleaching agent dosage.

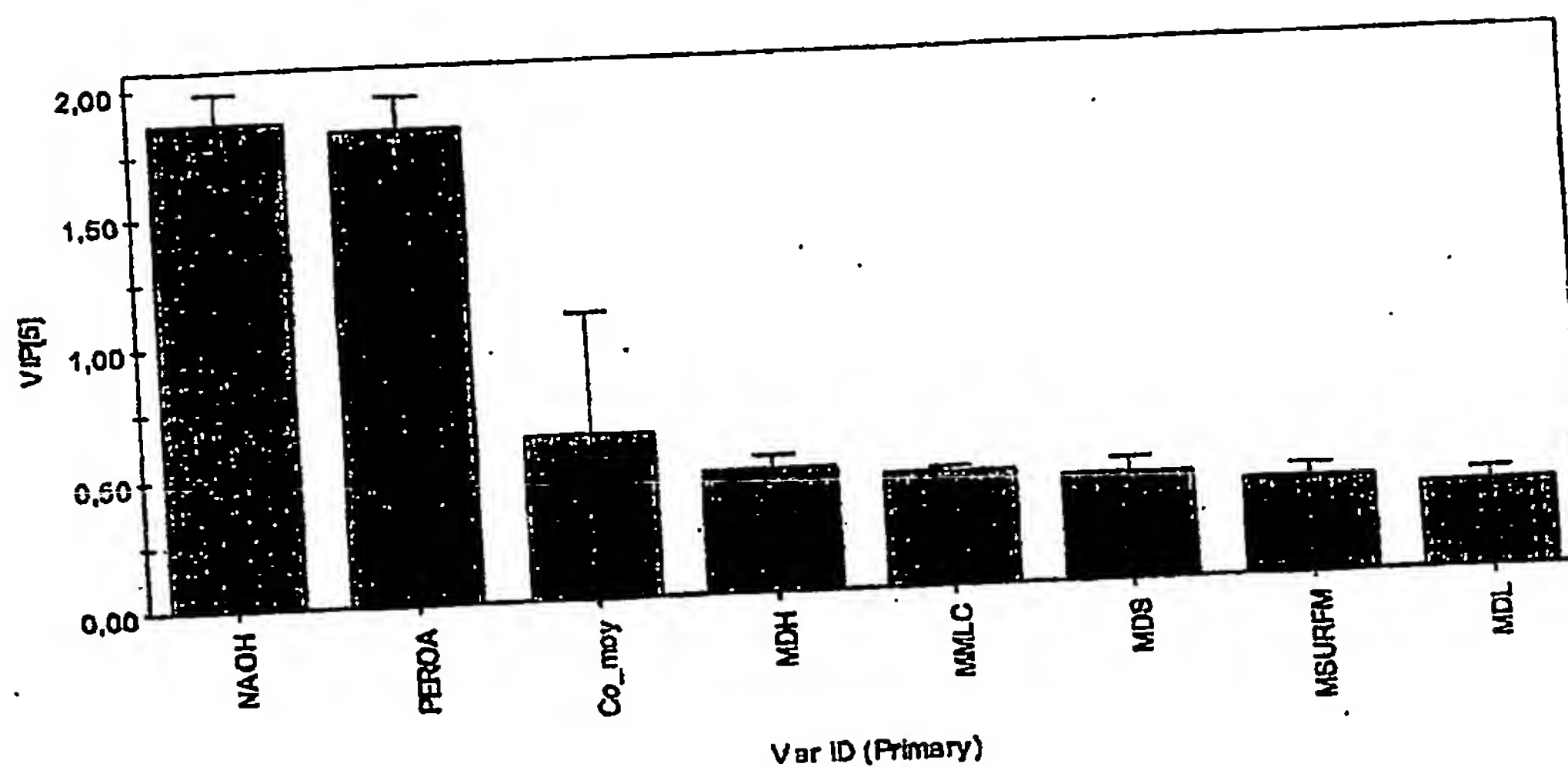


Fig. 1

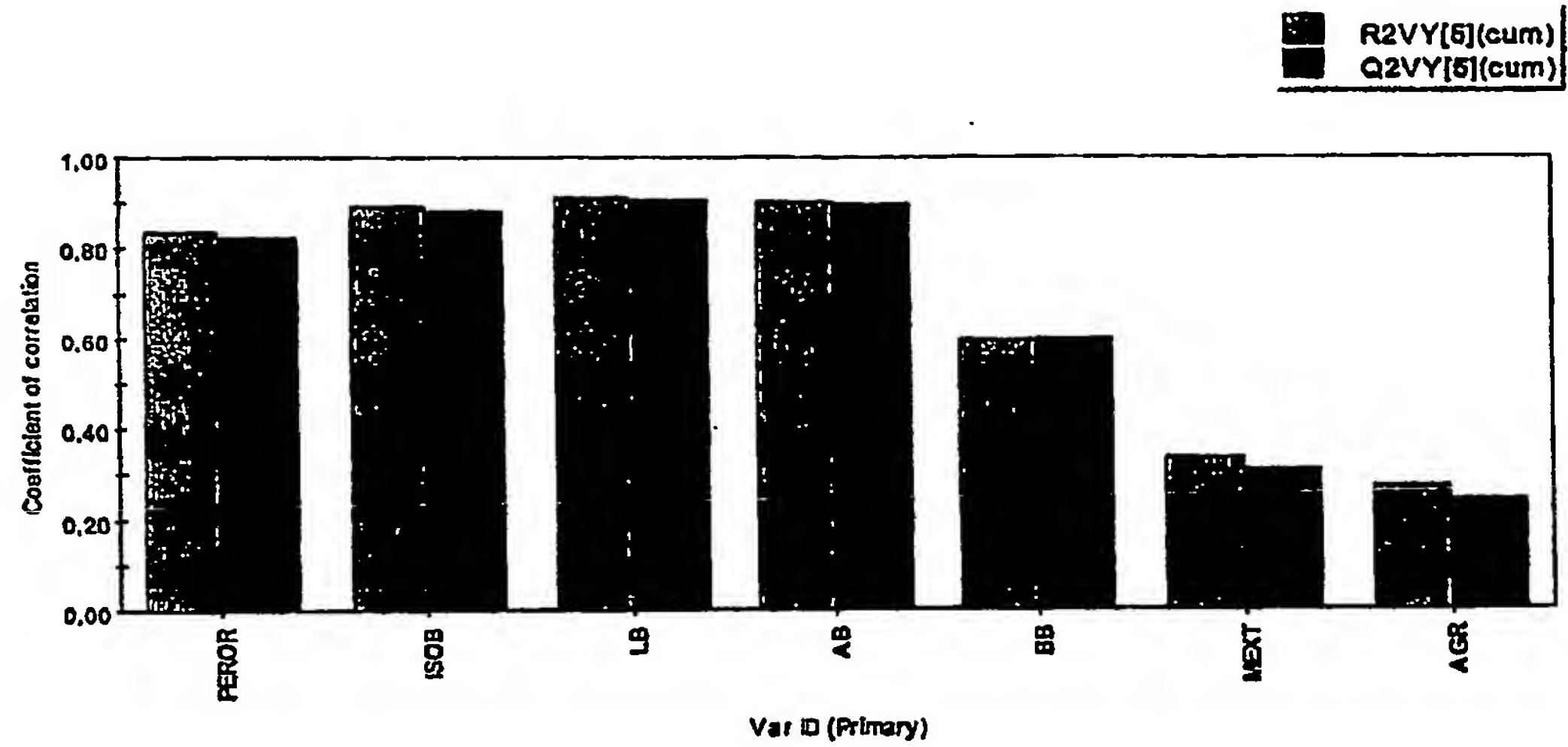


Fig. 2

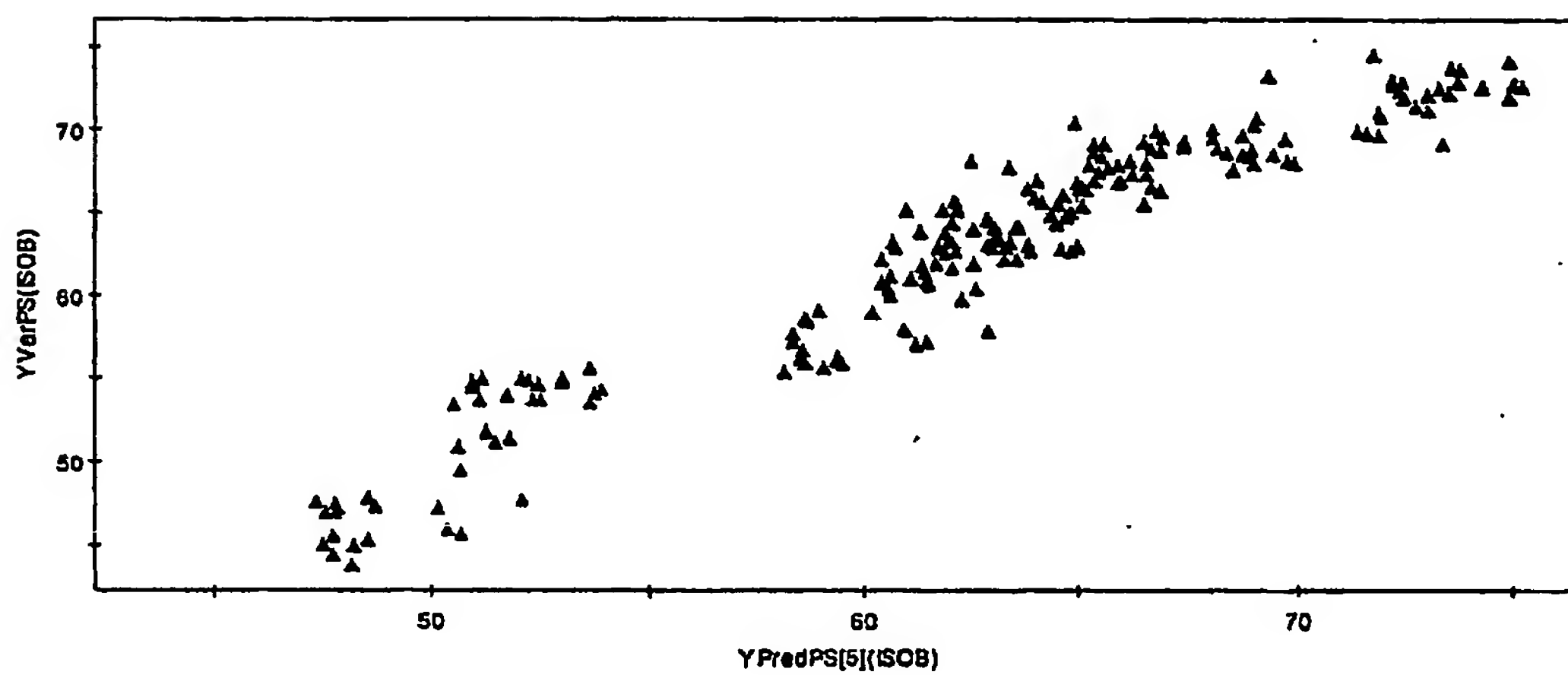
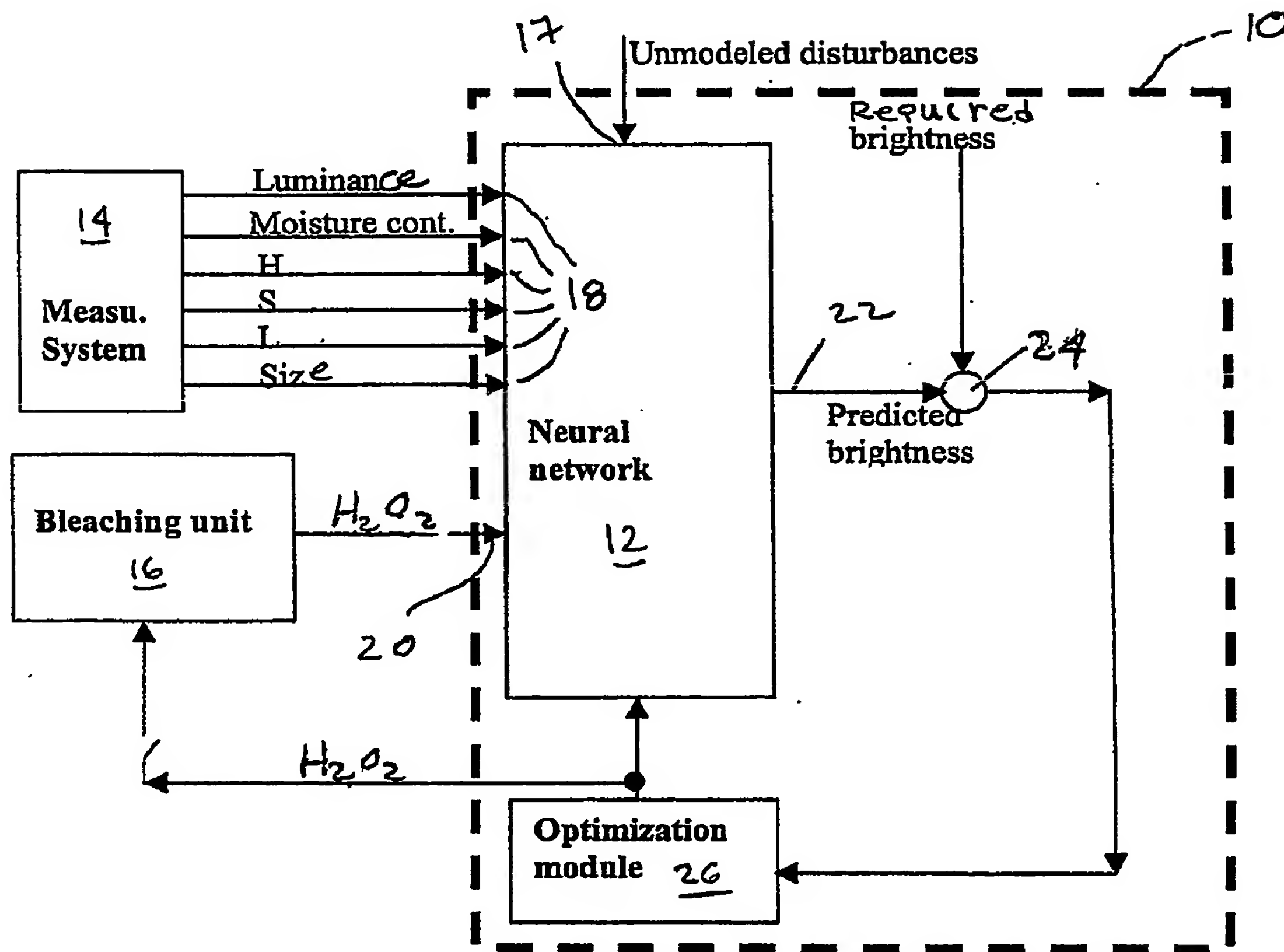


Fig. 3





**Fig. 4**

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**